A MINIATURE JOULE-THOMSON CASCADE LIQUEFIER CRYOSTAT FOR HELIUM

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ABSTRACT. A miniature cascade helium liquefier cryostat has been described and various constructional details given. The liquefier is designed to enable experiments at liquid helium temperatures to be performed in an ordinary laboratory and uses only liquid oxygen for the initial cooling. The liquid hydrogen needed to cool the helium below its inversion temperature is provided by a hydrogen circuit liquefying hydrogen continuously within the same machine. Compressed cylinder hydrogen is used avoiding the necessity of compressor or gasometer for hydrogen.

INTRODUCTION

The recent widespread interest in cryogenic research has resulted in the development of inexpensive small laboratory made liquefiers for helium, using liquid hydrogen externally or internally the three proprietory liquefiers of Collins (1947), Meissner (1957) and Weil (1955) being too expensive and not within the resources of most laboratories. Most of these cheap liquefiers therefore utilise the Joule-Thomson effect and the principal of regenerative cooling. The general principles underlying the practical design of the Joule-Thomson hydrogen or helium liquefiers are well-known (Croft 1961) and the construction of the usual liquefiers has been fully described (Ahlberg *et al* 1937, Rollin 1936, Ruhemann 1930, Starr 1941 Fairbank 1946, Daunt and Mendelssohn 1948, Chester and Jones 1953). For occasional work it is simpler and much less expensive to construct a double liquefier in which both hydrogen and helium are continuously liquefied, rather than set up separate liquefiers for hydrogen and helium. Thus starting only with liquid air, liquid oxygen or liquid nitrogen, a small quantity of hydrogen sufficient to obtain the necessary starting temperature for the helium stage is liquefied inside the apparatus itself. In the present paper a miniature Joule-Thomson Cascade liquefier for helium based on this principle is described, whose prototype is the liquefier of Chester and Jones (1953), but a few novel features have been employed. Further a few details of design and operation have been presented here which may be helpful to workers in this field particularly in this country.

Hydrogen gas of fairly high purity (99.5% or more) is commercially available in this country in cylinders at a pressure of 1980 lbs./in^2 and is quite cheap. Be-

cause of the relatively high cost of compressor-gasometer system, it is sound economy to use this commercially compressed gas for small scale liquefaction and to let off the low pressure hydrogen to the atmosphere. The standard 165 cu.ft. capacity cylinders at 135 atmospheres could be used down to 35-40 atmospheres. At our special request Messrs. Indian Oxygen Ltd. agreed to supply us Standard Purity (purged) locally manufactured hydrogen with impurities not exceeding 0.3% and this was available in any quantity. This greatly facilitated our work and involved very little cost.

GENERAL DESCRIPTION OF THE APPARATUS

A simplified diagram of the liquefier is given in figure 1. In common with most other inexpensive liquefiers, the hydrogen liquefaction circuit is fed from a manifold of high pressure hydrogen cylinders, and the hydrogen gas is subjected to

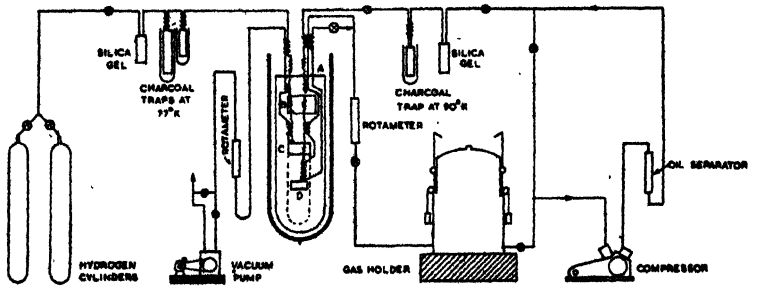

Fig. 1. Schematic diagram of the liquefier.

Joule-Thomson expansion in an open circuit. For the helium liquefaction circuit a small compressor is used, which delivers compressed helium at 40 atmospheres and this expands through the J. T. Valve in a closed circuit producing liquefaction.

The oxygen vessel B, the hydrogen vessel C and the helium vessel *D* are all made of brass and are suspended inside a vacuum case A, also of brass, which is attached by an easily demountable seal of Wood's metal. The vessel B is maintained at 62°K by constantly boiling liquid oxygen contained in it at a reduced pressure of about one cm. of mercury. The desired vacuum is produced with the help of Edwards 1S150 pump using tricresyl phosphate as the pump oil. An automatic valve actuated by metal bellows dispenses liquid oxygen via a gauze filter from the outer dewar to the vessel B and is of the same type as used by Chester and Jones (1953). The pumping speed was adjusted by a Saunders valve and the inflow of liquid oxygen in B was carefully adjusted by suitably fixing the rod which controls the valve seating. A general view of the assembly with the vacuum case removed is given in Fig. 2.

High pressure hydrogen and high pressure helium (25-30 atmospheres) are first cooled by the out-going low pressure hydrogen and helium in their respective

heat exchangers before entering the liquefier proper. The hydrogen exchanger is of special construction. The high pressure coils are wound round a rod and the

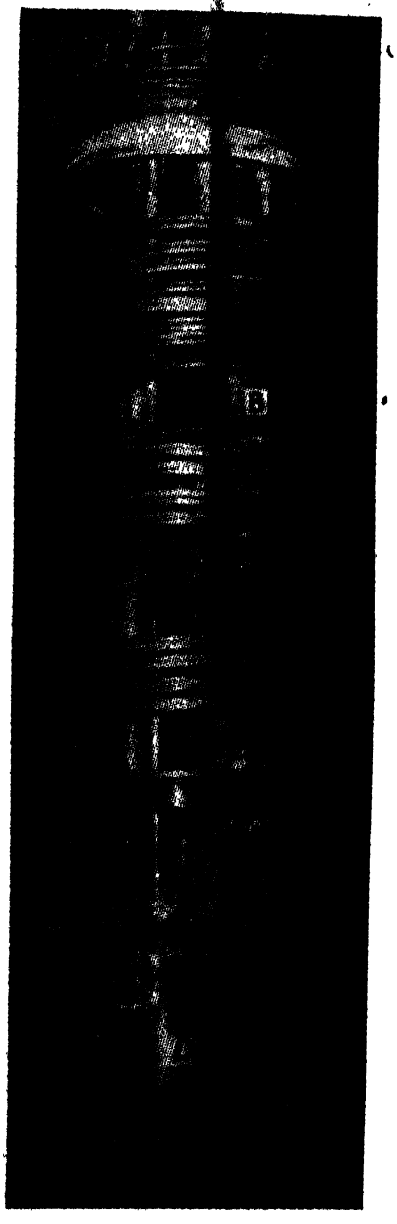

Fig. 2. General view of exchangers. (Photo with vacuum case removed.)

rod is inserted in a wide tube carrying the outgoing low pressure hydrogen. Next the high pressure hydrogen and the high pressure helium coil round in liquid oxygen in the dewar and finally enter the vacuum space. Here the high pressure hydrogen and helium coils are respectively solder-bonded to their low pressure

oils and these bonded coils of hydrogen and helium are arranged alternately nd coiled round the liquid oxygen vessel B but not soldered to it. Then the high ressure hydrogen and the high pressure helium coils are both soldered to the ower part of B and thus leave it at temperature of B. Next the high pressure ases are again cooled by the respective outgoing gases through solder-bonding n exchangers in the same manner as explained before. Then the high pressure ydrogen gas expands at the J.-T. Valve, where it ultimately liquefies and the iquid collects in the vessel C. The outgoing gas passes out through the various xchangers thereby cooling the incoming gas and is finally let out to the atmosphere hrough the rotameter so that the rate of flow of the gas can be measured. When he vessel C is filled with liquid hydrogen at 20°K, the hydrogen pump (Edwards S150) is started and the liquid allowed to boil at a pressure of 12 cm. of mercury roducing a temperature of 15°K.

The high pressure helium after leaving the exchangers traverses through oils soldered to the lower part of C. The helium gas thereafter enters the final xchanger at about 15°K and subsequently expands at the Joule-Thomson valve where part of it liquefies and collects in the vessel D. The vessel D is surrounded by a radiation shield which is in metallic contact with the bottom of the vessel D and thus the heat leak into D is minimised. The helium stage is also provided with a by-pass return path for helium in 1/4" tube, which in turn sheaths the control rod of the expansion valve; this tube can be used as a low resistance pumping tube to lower the temperature of liquid helium below 4.2°K.

A critical discussion of the performance of various types of heat exchangers has been given by Jacobs and Collins (1940) from which it will be evident that a highly efficient interchanger is expensive and difficult to construct. For ordinary purposes it is therefore usual to construct heat exchanges either in the form of concentric tubes or solder-bonded high and low pressure tubes. The latter design was preferred due to its simplicity of construction and two cupro-nickel tubes (H.P. tubes : 3/32 in o.d., 26 s.w.g, ; L. P. tubes : 1/8 in, o.d., 26 s.w.g. ; length 1-2m.) hard soldered together and suitably coiled were used.

All joints in the inter-changer systems were hard-soldered with "Easy-Flo" silver solder (Johnson, Mathey and Co., Ltd.). For reasons of safety the same solder was used for all other parts of high pressure line. For low temperature soft soldering, the solder used has only tin and lead (no bismuth) in the ratio of 40 Sn and 60 Pb.

HELIUM DISTRIBUTION AND PURIFYING CIRCUIT

Figure 3 shows the lay-out of the helium distribution system used in our liquefier. Chester and Jones used a small oil-free gas-tight compressor in a closed helium circuit which included a dry gas-holder in the form of a large rubber bellows. We have found that the ordinary oil-lubricated HCESE two-stage, single-acting

compressor (manufactured by Reavell and Co. Ltd., Ranelagh Works, Ipswich) originally meant for compressing air to 1000 lbs/in^2 with a capacity of 9 cu. ft.

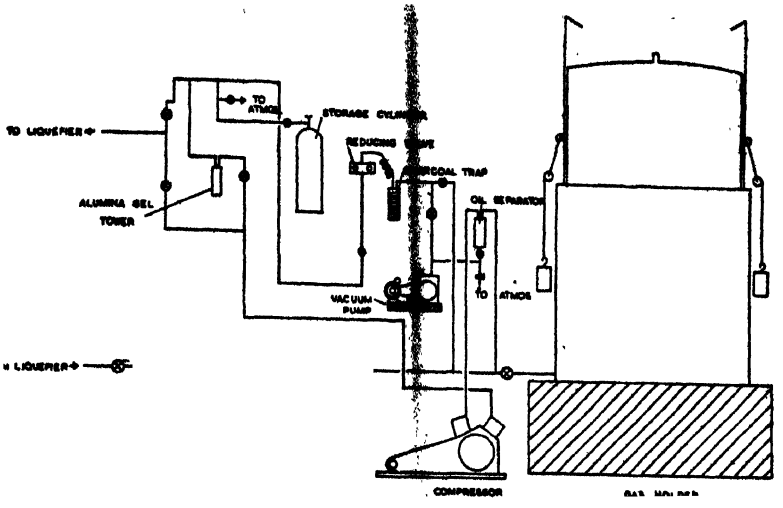

Fig. 3. Helium distribution system and Auxiliary purification circuit.

per minute when running at 1000 r.p.m. can be easily adapted for our purpose with slight modifications and is much cheaper. It is driven at 320 r.p.m. through V belts from a squirrel cage motor running at 950 r.p.m. and is capable of compressing 40 litres per min. of helium to a pressure of 40 atmospheres. It is fitted with enclosed relief valve on each stage with connections for piping back to the suction supply and a special seal at the point where the crankshaft passes through the casing so that there is no loss of helium. The usual rod for gauging the oil level in the crankcase has been replaced by a gauge glass and a special arrangement for adding oil without opening the crankcase to the atmosphere. Further the crankcase has to be joined to the gas-holder by tubing and the air hole is closed.

As the ratio of the two specific heats is larger for helium than for air, more heat will be produced during the compression of helium, and therefore a positive system of water circulation must be used to protect the compressing system from damage. For this reason our compressor has been fitted with an automatic switch designed (Sharma 1959) in this laboratory to break an electric circuit when the flow of liquid in the cooling pipe reaches a predetermined minimum.

The low pressure intake line to the compressor consists of a 1" copper pipe and connects it to the gasometer. The helium gas can be had from the gas-holder of 12 cu.ft. capacity. The dry gas-holders i.e. rubber bellows, as used by Jones and others are liable to develop leak and therefore a metallic gasometer supplied by Messrs. Tickford Ltd., is used and the gas is collected over transformer oil. Suitable alarm controls have been designed (Sharma 1961) and fitted to the gas-holder to indicate the maximum and minimum limits beyond which it is not be used.

The high pressure helium leaves the compressor through 3/8" o.d., 1/4" i.d. copper tubing and passes through an after-cooler of the conventional type i.e. the gas is passed through a copper tube immersed in water, followed by an oil separator in which the velocity of the gas is slowed down, so that it does not carry forward the oil and moisture. The compressed helium next passes to the distribution panel from which it can be sent to the liquefier, to the storage cylinders, or through a reducing valve, to the purification circuit and the gasometer as may be seen from figure 3.

As an added precaution for preventing the contamination of charcoal through oil vapours, the dry helium gas from the distribution panel is [further allowed to pass through a porous ceramic cylinder of pore size 7.5 to 10.0 microns which serves to remove any traces of oil. Finally a silica gel tower is used to remove completely any moisture from the gas. An alumina gel tower is inserted between the distributing panel, and the storage cylinder for removing oil and moisture, so that the helium stored in the storage cylinders is dry and oil-free.

Auxiliary purification Circuit. Before starting a liquefaction run, the helium in the gas holder should be carefully purified. This is done with the help of the auxiliary purification circuit. First all the gas from the gas-holder is transferred by the compressor to the storage cylinders, and from there it is passed through a reducing valve which reduces the pressure to about 4 atoms. Thereafter the gas goes through the charcoal trap C kept immersed in liquid oxygen, so that it is purified/by the activated charcoal and finally the purified gas collects in the gas holder. This process is repeated three times before every liquefaction run. It may be mentioned that B.O.R. 13 (supplied by Indian Oxygen Ltd.) which gives an outlet pressure ranging from 0 to 200 lbs/sq. in is quite convenient to use as a reducing valve in this circuit and has been used by us for this purpose.

OTHER CONSTRUCTION DETAILS

Charcoal traps. The charcoal pot is made from vibrac steel bottles supplied by Messrs. Vickers Armstrongs. It is packed with about 500 cc. of activated charcoal supplied by M/s. British Carbo-Norit Union of grade ULTRASORB S. C. 11. The open end (i.e. lower one) is sealed with screwed plugs of the same material and soft-soldered in place. This allows for easy refilling when necessary. A filter inserted in the charcoal vessel prevents particles of charcoal or dust being carried further by the gas streams. The incoming and the outgoing lines support the pot and are joined to the circuit by detachable unions. The finished bottle is hydraulically tested to twice the working pressure when it should show no distortion.

In our experiments using locally manufactured hydrogen the J-T valve occasionally got choked showing that the degree of purification attained was not sufficient. We, therefore, inserted an additional charcoal pot of capacity about 400 cc. Using both of these in series there was never any trouble of blocking.

Porous Ceramic Cylinders. Porous ceramic cylinder (Aerox Ltd., U.K.) of porsilex Grade P.35 filter elements, 10" long × 2" o.d. × 1½" i.d., open at both ends were used for removing oil from gaseous helium at 40 atmosphere pressure. The filter casing is made of copper and is suitably mounted in the high pressure line of helium. The ceramic element is easily replaceable by removing the body of the filter unit from its head by releasing the retaining bolts, and then unscrewing the locking nuts and cap.

For Low pressure line purification. Wire gauze filters packed along the tube are generally used but this reduces the suction capacity of the compressor. Activated alumina also has the same disadvantage. We have, therfore, employed a simple oil separator made out of ordinary copper pipe length 10½", 2"o.d. 1⅞" i.d. inside which are mounted several fins on a vertical axis. The whole is sorrounded by ice and the oil vapour, if any, condenses and can be drained off. This is, however, not very essential as we are not using an oil free compressor and any oil vapour carried forward from the gas holder is not likely to do any great harm.

Joule-Thomson Valves. Unlike the apparatus of Chester and Jones we have used an adjustable expansion valve in the hydrogen stage which is further provided with a locking device as a precautionary measure to prevent excessive opening of the valve. This allows us to adjust the rate of flow of hydrogen and is useful at the time of blocking of the valve. The helium valve is also adjustable. These valves are the usual type of needle valves with long stem and seat. The top part of the valve is threaded and can be easily unscrewed and withdrawn. The two tubes sheathing the control rods are provided with relief valves at the upper end; this protects the hydrogen and helium liquid vessels from any high pressure accidentally developed. The valves are so constructed that the whole valve including the seating, can be withdrawn after only two simple soldered joints have been broken.

Apart from the Joule-Thomson valves already mentioned, the high pressure valves used are the M.K. VIII (M/s. Nico-light) valves while the low pressure valves are the Saunders valves with vacuum reinforced T and S grade

INDICATORS

Gauges are permanently mounted to measure the intake pressure of cylinder hydrogen and compressed helium, and the pressure on the high and low pressure sides of the Joule-Thomson valve in both circuits. The temperature of the liquid hydrogen can be measured by the hydrogen-thermometer acting as a vapour thermometer and the temperature of helium vessel is measured by means of a

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INDICATORS

Gauges are permanently mounted to measure the intake pressure of cylinder hydrogen and compressed helium, and the pressure on the high and low pressure sides of the Joule-Thomson valve in both circuits. The temperature of the liquid hydrogen can be measured by the hydrogen-thermometer acting as a vapour thermometer and the temperature of helium vessel is measured by means of a

helium thermometer, acting either as a gas thermometer or a vapour pressure thermometer.

Rotameters, with duralumin floats supplied by Rotameter Manufacturing Co. are suitably inserted in the low pressure lines to measure the rate of flow of the gas in each circuit.

For check on the vacuum in the metal case A jacketing the liquefier unit, a Cathedon combined Pirani and Ionisation gauge (W. G. Pye and Co.) was mounted with both probes inserted. This served as a rugged and satisfactory indicator throughout the range. A vacuum of the order of 5×10^{-6} mm. of mercury was maintained inside the casing by a fractionating oil diffusion pump (*F* 203, W. Edwards and Co.) and thus a good thermal insulation was obtained.

HELIUM EVAPORATION CIRCUIT

For attaining temperatures lower than 4.2°K, it is necessary to evaporate liquid helium under reduced pressure. For this purpose a parallel circuit is provided for pumping the liquid helium in D through the helium by-pass tube and the helium gas going out of the pump is taken through an oil separator to the gas holder, the direct connection to the gas holder being closed. If desired, the degree of vacuum in D is controlled by pumping through a micrometer valve of Nagretti and Zambera type.

OPERATION AND PERFORMANCE

Preparatory to a run, the charcoal pots and other high pressure circuits of the liquefier are carefully checked for leaks under the highest operating pressure. Next the hydrogen and the helium charcoal cleaners are kept at about 200°C for at least two hours and pumped continuously. During this reactivation of charcoal the bottom plugs of the cleaners are kept moistened to prevent the soldered joints from softening. Heating is then stopped and when the system has cooled, the pumps are turned off and the high pressure gases admitted to their respective systems.

Just before putting the plant in operation the low pressure and high pressure lines of both the hydrogen and the helium circuits are evacuated by their respective pumps and the charcoal cleaners are surrounded with liquid oxygen. Each system is then flushed with the respective gas purified by charcoal cooled by liquid oxygen. The process of evacuation and flushing is repeated several times and finally the liquefier is filled with pure gas in each system. Next the outer case A of the liquefier is connected to the rotary pump (the diffusion pump is kept ready) and after about ten minutes hydrogen exchange gas is introduced into the vacuum case. Then it is surrounded with liquid oxygen in a glass

dewar. The approximate timings of subsequent events in a typical run is as follows :

Time	
0 min.	Dewar vessel containing liquid oxygen is put on to the apparatus and the circulation of helium gas (30 litres/min) started with the helium by-pass open.
15 min. :	At this instant the temperatures of B, C and D will be 90°K. Liquid oxygen pump is started. Also high vacuum pump for evacuating the case started.
25 min. :	Vessel B at 62°K (Helium flow is continued). Hydrogen flow (30 litres/min) also started.
30 min. :	Vessel C at 62°K and D a little higher. Helium flow stopped.
37 min. :	Liquid hydrogen in C. Hydrogen pump started and the helium (15 litres/min) also started through the helium heat inter-changers with by-pass closed.
42 min. :	Hydrogen flow is reduced to 10 litres per minute and helium flow is continued as before.
55 min. :	Liquid helium in D.

The only departure we made from Jones procedure was that once liquid hydrogen had been formed and hydrogen pump started, the helium circulation was re-started with the by-pass closed rather than open. This slightly lengthened the cooling time, but is thermodynamically more efficient. Another advantage of this procedure is that there is no risk of liquid hydrogen completely evaporating in vessel C as the helium flow is kept down.

When once the initial difficulty of handling the apparatus had been overcome, it was found possible to maintain helium temperatures steadily for several hours. By boiling liquid helium at a reduced pressure of about 50 cm. a temperature of 3.25°K was attained and maintained for several hours. Only three cylinders of 165 cu.ft at 1980 lb/in^2 and 25 litres of liquid oxygen were necessary for a run of about 5 hours at a temperature 3.25°K. For maintaining a temperature of 4.2°K for several hours one to two cylinders of hydrogen and about 15 litres of liquid oxygen would be sufficient. The rate of production of liquid helium in our apparatus is about 2.8 cc/min. with an efficiency of about 0.13 which is about 87% of the theoretical yield.

The construction and installation of the liquefier was mainly due to the inspiration, encouragement and foresight of the late Professor M. N. Saha, D.Sc., F.R.S. One of the authors (B.N.S) expresses his grateful thanks to the late Professor Sir Francis Simon, Kt., O.B.E., F.R.S. and his colleagues of the Clarendon Laboratory, Oxford, for their kind hospitality and co-operation during his stay in

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